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Environmental Laboratory Sample Acceptance Policy

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Record of Revision

| Revision | <u>Date</u> | Responsible Person | Description of Change |
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| 0 | 11/18/01 | James Adamski | Initial Release of Document |
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| 3 | 05/05/09 | James Adamski | Air Sampling and Asbestos Deleted Holding Time Specifications updated Membrane Filtration Section Added Transporting infectious materials added Section on trip blanks added |
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Record of Annual Review

| Name (Printed) | Title | Signature | Date |
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I. Introduction

As a certified environmental testing laboratory, the Nassau County Department of Health, Division of Public Health Laboratories, is committed to the objective of producing environmental data that is technically defensible, scientifically valid and comparable to data generated nationwide by other laboratories. In order to accomplish this goal, national laboratory standards and protocols specified by "NELAC" (National Environmental Laboratory Accreditation Conference) must be rigorously followed. Recognizing that there is a serious and considerable responsibility involved with providing analytical data that will be used to determine the extent of compliance with environmental regulations, laws and standards, and the subsequent adverse economic impacts environmental violations and remedial actions may have, the Nassau County Department of Health has set up and formalized a "Sample Acceptance Policy" consistent with "NELAC" data quality objectives that clearly defines procedures, protocols, criteria and circumstances under which environmental samples will be accepted for laboratory analysis.

The intent of this document is to provide instructional reference material for personnel engaged in the collection of environmental samples and users of environmental data so that they can better understand and appreciate the requirements of NELAC. A major premise of environmental testing is that samples must truly be representative of their sources. As a result, the collection process itself has an important influence on the outcome of testing and the interpretive value of test data. Since the techniques used in sample collection, preservation and transportation have a significant impact on whether the sample is truly representative of conditions at the exact time of sampling, they accordingly have the potential of causing a biased test result when not performed properly. All aspects of sample handling must be considered as an important integral component of the analytical process.

II. The Sample Collection Process

A. Sampling Site Selection

During any type of environmental investigation, the choice of sampling locations will have a major impact on its outcome. The location of sampling sites should be planned in advance and chosen to fulfill specific objectives. If the adherence to specific water quality standards are a component part of the investigation, procedural guidelines for sample collection may already be available and should be appropriately followed. If limits have been incorporated into local ordinances or regulations for specific contaminants, sample collecting activities must conform to any pre-established legal requirements. The following summary offers some general guidance in selecting representative sampling sites, but every environmental investigation is uniquely different and it is oftentimes necessary to creatively adjust or customize a sampling program accordingly.

1. Public Drinking Water

When conducting a distribution system survey, select collection points that are truly representative of the water supply. Sample from taps that are connected directly to service mains and avoid taps that are connected to cisterns or storage tanks unless these sources are to be considered a representative segment of the distribution system. Choose taps that are dry and in good physical condition. Highly corroded taps that are leaking or moist are a potential source of contamination. When sampling a supply well, choose a tap on the discharge side of the mechanical pump and as near to the pump as possible. If the well has no pumping machinery,

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collect the sample by using a weighted container. When water remains stagnant in a well for extended periods of time, surface scums may form and become a potential source of contamination. Purposely monitor sites in a distribution system that may become potential problems. Sites near dead-end sections may contain higher levels of contamination because of reduced flow. Close proximity to cross connections, breaks in a distribution lines or areas of reduced line pressure may increase the possibility of contamination. Pay attention to the sanitary conditions at the sampling site. If commercial establishments, private residences or public buildings are used as sampling sites, the lack of cleanliness increases the possibility of contamination.

2. Surface Waters

Lakes, rivers, streams, estuaries, tidal flats or other bodies of water may be routinely monitored to determine the impact of domestic wastewater, industrial discharges or storm water runoff on overall water quality. These monitoring programs should include sites representing baseline or locations that are not impacted, raw discharges where contamination levels would be at a maximum and a series of sites in the receiving waters that will adequately characterize the dispersion of wastewater from the points of discharge. Collection sites should be selected to define variations in contaminant levels with respect to time, water depth and distance from the points of discharge. The dispersion plume profile defined by these variables will form a basis for evaluating environmental impact. Sampling frequency must be adjusted to adequately define seasonal changes, diurnal changes, water usage, storm events, tidal fluctuations or other cyclic phenomena that may superimposed upon a wastewater distribution model. When sampling off the side of a boat, collect samples from the upstream side. Boat engines should be turned off during sampling to avoid contamination from engine exhaust. When sampling from a bridge, sample in open waters well-removed from pilings or other supporting structures that could modify flow patterns. Avoid collecting samples in shallow water too close to shorelines. Shallow water is easily fouled by turbulent mixing with bottom sediments.

Beach areas used for recreational bathing should be sampled in a manner that defines water quality for the entire recreational zone. Define the quality of water in swimming areas by collecting samples at a uniform depth of approximately one meter. The full impact of recreational activities cannot be determined without first defining the characteristic changes associated with naturally occurring environmental phenomena. Use sampling points in peripheral areas outside of the recreational zone to establish normal background levels. Choose a sampling frequency that reflects the full spectrum of water conditions. Sample at times where both the maximum and minimum recreational usage are best represented. Sample at times associated with low, ebb and high tides to determine the effects of tidal flushing. Finally, collect additional samples as necessary to characterize the frequency and intensity of storm events and the impact of runoff water from these events.

3. Sediments

Bottom sediments may be used as a stable index of water quality. Changes in sediments are more indicative of long term environmental processes and may be used to better understand how the overlying body of water may be changing. Sediment behavior depends on the temperature of the overlying water and goes through natural seasonal changes. These natural changes must be carefully considered when interpreting testing data. Choose sampling sites that are well removed from shorelines. Sediments in shallow areas can be disturbed by wind turbulence. Sediments in shallow areas can also be influenced by runoff water during storm events. Any physical disturbance of a sediment during the sampling process may cause erratic testing data. There are

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numerous types of mechanical devices available for collecting sediment samples. Choose the sampling device that is recommended for the analytes being tested.

4. Soils

Natural variations in soil composition make the interpretation of analytical data extremely difficult. Collect samples that have a uniform matrix. Choose sites that do not contain large pebbles, chunks of stone, vegetation or other foreign debris as these can potentially skew the test results. A sufficient number of samples must be collected to determine natural variability in a baseline area before attempting to define variability from a suspected contamination source. Choose sampling areas rather than specific points and collect several samples in each designated area to form analytical clusters. Attempt to establish levels of reproducibility within each cluster before making any interpretive judgments. Use valid statistical methods to establish trends or define concentration gradients above background levels. The analytical difference between cluster samples is a more valid basis for comparison than that of individual samples.

B. Representative Sampling

No sampling method is perfect. Changes can and will take place regardless of the effort made to prevent this. One can only attempt to minimize these changes as much as possible. Sampling programs should be carefully designed to obtain samples that will not deteriorate, become contaminated and/or otherwise compromised before analysis. All handling and transport activities must be constantly evaluated to prevent, or at least minimize, any significant changes in sample composition from occurring prior to testing. Factors that are capable of altering a sample may include, but are not limited to the following:

- 1. Chemical reactions between the sample and container walls, closures, seals and/or liners
- 2. Chemical interactions between sample constituents
- 3. Degradation of sample constituents by biological processes such as the metabolism of microorganisms, enzymes released from lysed cells or biomass decay
- 4. Chemical degradation of unstable analytes
- 5. Artificially induced increases in the concentration of an analyte due to the presence of chemical precursors in the sample matrix
- 6. Artificially induced increases in the concentration of an analyte by cross contamination mechanisms (e.g. diffusive transport from air to water)
- 7. Loss of volatile analytes
- 8. Photochemical (light induced) reactions
- 9. Equilibrium reactions effected by changes in temperature and/or pressure over time
- 10. Adsorption or ion exchange processes on container and/or closure surfaces.

C. Sample Stabilization Techniques

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Many of the dynamic processes that could change the composition of a sample during or after the collection process can be prevented or significantly retarded through the use of prescribed techniques. Although the intent of these techniques is to stabilize samples and ensure "representativeness", it is important to note that these measures are many times analyte specific so that a stabilization technique for one analyte may be totally inappropriate for another. In many cases, the use of an incorrect sampling technique may totally invalidate a sample. Some of the stabilization techniques may include, but are not limited to the following:

- 1. Use of inert, analyte compatible sampling containers and closures
- 2. Proper tightening or sealing of closures
- 3. Exclusion of light
- 4. Exclusion of air bubbles from aqueous samples
- 5. Lowering sample temperature
- 6. Use of preservation agents as additives
- 7. Adjustment of pH
- 8. Elimination of chemical precursors
- 9. Minimizing transit and storage times
- 10. Removal or avoidance of cross contamination sources

A sample's suitability for acceptance is greatly dependent on the treatment a sample receives prior to its arrival at the laboratory. Cooling a sample to a temperature between 2 and 6 degrees Celsius is the most common method of sample preservation. Laboratory personnel record sample temperatures when they arrive at the laboratory and this is used as an important criteria for sample acceptance. When the holding time is very short, samples may not have had sufficient time to reach the required holding temperature. Laboratory personnel shall accept samples in this condition as long as there is evidence that the cooling process has begun (e.g. arrival on ice).

All sample containers for bacteriological assessment must contain sufficient sodium thiosulfate to ensure the removal of residual chlorine as interference. Bottles acquired from the Division of Public Laboratories will have the sodium thiosulfate already added to the container prior to sampling in either powder or pellet form. Acid-preserved samples are acceptable if a test with pH paper or a pH meter yields a result of <2. All exceptions to sample submission protocols will be appropriately recorded at the time of submission.

Composite samples are sometimes used to represent periods of time, depth profiles or an assembly of sampling points. In such instances, the details of collection will vary with localized conditions, so specific procedures for the preparation of a composite sample are not universally applicable. The use of composite samples is not generally recommended. It is more informative to analyze a number of separate samples instead of one composite so that variability, maxima and minima can also be determined. The major disadvantages associated with composite samples include loss of analyte relationships, potential dilution of analytes below detection limits, an increased potential for analytical interferences and an increased possibility of analyte interactions. It is more scientifically correct to

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prepare a statistical composite based on the data obtained from individual samples than to analyze a composite sample. The notable exception is when a regulatory agency specifies a composite sample in its permit requirements. The use of BOD data in evaluating the performance of waste water treatment facilities is such an exception.

Fill sample containers without pre-rinsing with sample, unless there are analyte specific instructions to the contrary. Pre-rinsing will result in the loss of any pre-added preservatives and may cause a chemical interaction between the sample and the container surface to produce a positive or negative bias for some analytes. If a known preservative has already been added to a container, take care not to overfill the container, as the preservative may be lost or diluted. Except when sampling for volatile organics or dissolved gases, leave an air space equivalent to approximately one percent of the container's total volume to allow for thermal expansion.

A laboratory representative shall consult with the sample collector or appropriate authority regarding the nature of any improper submission requirements and discuss the potential impact this will have on the status of the sample. The laboratory shall maintain records concerning any correspondence and/or conversations regarding the final disposition of a sample.

D. Trip Blanks

Analytical blanks are artificially prepared samples made from high purity distilled and/or deionized water known to be free of all the possible analytes that may be found in an environmental water sample. The blank is subjected to the same analytical and measurement processes as real samples in order to establish a zero baseline or background value that will verify the validity of test results. Trip blanks are a special type of analytical blank or field blank specifically set up to monitor for contamination that may be introduced during the sample collection, transport and storage process. Some analytes such as coliform organisms and volatile organic compounds are more prone to contamination than others, but any analyte could be introduced as a contaminant under the right conditions. Trip blanks are carried in coolers, stored on ice and subjected to the same forms of chemical preservation just like real samples and will be exposed to the identical conditions as real samples during the course of a work day. In the event that contamination is detected in the trip blank, the validity of all the samples associated with that trip blank become questionable. Data from trip blanks have proven to be extremely useful in tracking down sources of contamination and have played a major role in improving the quality of the sampling process. Trip blanks are especially useful when evaluating public water supplies and recreational beach areas for bacterial contamination by making it easier to identify false positive findings.

E. Hazards Associated With Sample Collection

Sample constituents may potentially pose a number of health related risks to personnel engaged in the collection of environmental samples. Sample constituents may be toxic, flammable, corrosive, radioactive and/or infectious. These constituents can enter the body through the skin, eyes, lungs, mucous membranes or by oral ingestion. Food may easily be contaminated with these constituents either be direct contact with hazardous samples or indirectly by adsorption of hazardous vapors. Sampling personnel must plan their activities to avoid situations that lead to accidents or unnecessary risk. Some of the general safety rules to follow during sample collection include, but are not limited to the following:

- 1. Never leave food near samples or near sampling locations.
- 2. Do not eat or drink near samples or near sampling locations.

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- Always wash hands thoroughly before handling food and immediately after sample collecting activities.
- 3. Do not smoke or use any equipment that could potentially generate sparks, flames or excessive heat near samples or sampling equipment, especially if the presence of flammable materials or vapors is possible.
- 4. Wear protective apparel that is appropriate to any suspected hazard. Precautions may be limited to wearing gloves and eye protection, but may also include special coveralls, aprons or hazard suits.
- 5. Eye protection is recommended during all types of sample collection activities. It must be made of safety glass or plastic and include side shields.
- 6. If the presence of toxic or flammable vapors are known or suspected, sample only in well ventilated areas or use an appropriate respirator or self-contained breathing apparatus.
- 7. Adequately label samples with appropriate warnings whenever there is a suspected hazard. This may be due to flammability, corrosivity, toxicity, oxidizing materials, radioactivity or the presence of an infectious biological agent. This will ensure that any other personnel engaged in the subsequent handling, transport, storage, testing and disposal of these samples are appropriately notified of their potential hazards.
- 8. If there is any doubt as to the level of protection or the types of safety precautions necessary during specific types of sample collecting activities, consult with a knowledgeable industrial hygienist or safety professional.

III. General Protocols for Environmental Samples

The laboratory assumes responsibility for the quality control of sampling containers, sampling kits, preservatives or special collection apparatus. These items shall not be obtained from any source other than the laboratory performing the analysis unless written permission is provided. An appropriate qualifying statement to this variation shall be included on the final laboratory report.

Sample collection shall be performed using NELAC approved materials and protocols. Parameter specific requirements defining the proper containers, volumes of sample, preservation techniques, holding times and special handling instructions are provided in Appendix A. Prompt delivery to the laboratory immediately following the collection of samples is strongly recommended but not a requirement for samples that have been properly preserved. The risk of contamination is significantly reduced when the holding time prior to laboratory delivery is as short as possible. If an environmental sample deviates from NELAC acceptance requirements, testing data on this sample shall be "qualified" in an unambiguous manner that clearly defines the nature and circumstances of the deviation. Only personnel who have been properly trained in the collection of environmental samples shall be authorized to perform this activity. The laboratory shall not accept samples from individuals who do not have the appropriate qualifications for this assignment.

If samples must be collected during irregular hours such as at night, weekends or holidays, a special holding room is available for use by Health Department, Fire Department or law enforcement personnel. This room has its own locked entrance and provides a safe and secure location for the storage of environmental samples until they can be formally accepted for analysis by laboratory staff. It is available for use 24 hours a day, seven days per week. This room is equipped with sampling

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supplies, containers, preservation reagents, ice packs, coolers, refrigerators and freezers.

Samples arriving at the laboratory are each assigned a unique identification code known as the "Laboratory Accession Number". This number becomes the primary means of identification and tracking throughout the testing process. This procedure formalizes the submission of a sample and establishes an unambiguous link between all sample containers and all of the analytical records associated with these sample containers. "Laboratory Accession Numbers" are assigned in chronological order as they are received at the laboratory and a permanent record of this process is maintained in an accession log book. Two logs are maintained for samples brought to the laboratory The first is a "Receiving Log" that documents the transfer of samples from field personnel to laboratory personnel. The second is an "Accession Log" that allows the sample to be tracked through every subsequent step of the laboratory analysis. This "Accession Log" must minimally include the following information.

- A. The Laboratory Accession Number
- B. The field ID code assigned at the time of collection
- C. A source description
- D. Date and time received at the laboratory
- E. The initials or signature of the laboratory employee who has officially received the sample as a representative of the laboratory.
- F. An indication that the sample was acceptable for analysis
- G. Qualifying statements for any deviation from the acceptance protocols.

The Division of Public Health Laboratories is required by NELAC to maintain all records and data that will allow the complete reconstruction of an analytical process culminating in a final result.

IV. Protocols for Proper Documentation

Samples must be submitted with field documentation that is legible and complete. Forms for this purpose are available from the Division of Public Health Laboratories. The field information on the sample submission form must include, but is not limited to the following:

- A. Sample identification code (field number)
- B. Location of the collection site (address, etc.)
- C. Site description
- D. Date and time of collection
- E. Name of person collecting the sample
- F. Method of preservation if applicable

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- G. Sample type and/or relevant commentary
- H. Names of the analytes for testing (analyte group name)

Labels used for identifying sample containers shall be durable (water proof). Information recorded on container labels must be clear and unambiguous using indelible ink. Sample container labels must contain sufficient information to establish an accurate and verifiable link between the container and its accompanying documentation forms. The labels on sample containers shall minimally include an initial sample identification code (field number), but additional information such as site name, collection date and time are helpful in establishing an unambiguous link between the container and its documentation.

V. Acceptance for Analysis

When samples arrive at the laboratory, technical staff must determine the sample's suitability for acceptance. Any departures from standard sample submission protocols must be recorded during the accessioning process. Any decision to proceed with an analysis not meeting the acceptance criteria must be documented and the data formally "Qualified" on the final laboratory report. Records or notes relevant to any correspondence and/or conversations with sample collectors regarding the decision to accept or reject a sample shall become a permanent part of the laboratory's record on that particular sample. In cases where the validity of a sample has been compromised to the extent that the interpretive value of the resulting analysis would be meaningless, laboratory staff shall have the option of not accepting the sample for analysis. Typically, a sample is found unacceptable for one or more of the following reasons:

- A. The identifying information on the container label does not match with the information on the accompanying documentation.
- B. The sample container is damaged, cracked or leaking
- C. There is physical evidence of improper storage and/or transit conditions (e.g. excessive heat or cold)
- D. An improper preservation agent and/or technique was used
- E. There is visible evidence or high likelihood of contamination
- F. Labeling is illegible, smeared and/or indecipherable
- G. The sample has been held beyond the accepted holding time
- H. The Chain of Custody time line is not continuous

VI. Chain of Custody Requirements

In the event that an environmental sample is intended be used as legal evidence in a court of law, NELAC (National Environmental Laboratory Accreditation Conference) has established formal Chain of Custody (COC) protocols to be implemented by field and laboratory personnel. The basic premise of

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a legal chain of custody record is to establish an intact continuous written record of physical possession (including storage and disposal) of environmental samples for their entire existence. The COC must accurately account for all time periods associated with a sample. This means that a time line has to be prepared demonstrating possession and exchange of possession at every step along the time line with no breaks. This not only applies to the original sample, but any additional aliquots, extracts or digestates that have been prepared from the original sample. By NELAC definition, a sample is in someone's custody if:

- A. It is in one's physical possession
- B. It is in one's view, after being in one's physical possession
- C. It is first in one's physical possession and then locked up to prevent tampering.
- D. It is kept in a secure area which is restricted to authorized personnel.

The COC records must include signatures of all individuals who had possession and access to a sample. Efforts should be made to simplify this record keeping process as much as possible by limiting the number of people who physically handle a sample. It is acceptable for the laboratory to designate a sample custodian, who shall be responsible for the receipt, storage and distribution of the sample to the appropriate work stations in the laboratory. Ideally, the COC record shall be limited to a single form that shall remain with the sample during transport, shipment and storage. It is recommended that the starting point for legal chain of custody be the time at which clean sample containers are distributed to field personnel at the laboratory. This has the advantage of adding another level of credibility to the collection process since a short time line between container distribution and sample collection is a convincing argument for establishing minimal risk from contamination. NELAC, however, also states that in most cases, it is acceptable that the legal chain of custody begin at the time of sample collection.

Samples delivered to the laboratory during normal business hours will undergo an exchange of custody by formal declaration on a COC form. This involves the person transporting the sample to the laboratory formally relinquishing the sample by signature to the laboratory custodian or other designated laboratory representative who then countersigns the COC and officially accepts possession of the samples. The samples must remain in the laboratory custodian's possession until the samples are properly accessioned, logged and labeled. Each transfer or handling procedure must minimally include the two signatures of the participants in the custody exchange, the time of day to the minute and the calendar date of the exchange. Notes regarding the purpose of the custody exchanges during collection, sample receipt, preparation, analysis and reporting should be included to produce unequivocal accurate record for all activities associated with a sample. The Nassau County Department of Health Laboratory provides a special chain of custody record form designed to accompany all sample submissions.

Chain of custody documentation must include the following:

- A. The same identical information that appears on the sample label.
- B. The name and address of the person(s) or organization(s) authorized to receive the laboratory report.
- C. The name of the person collecting the sample and a signed statement by this person attesting that the sample was collected using prescribed protocols.
- D. The signature of the person who has released custody and the person who has accepted custody during each custody exchange.

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- E. The calendar date and time of the custody exchange recorded to the nearest minute.
- F. A brief comment explaining the purpose of the custody transfer.

Once samples have been received at the laboratory, laboratory personnel will become responsible for the care and custody of the samples and will be prepared to testify that the samples were in their possession for the remaining lifetime of the sample. The laboratory will be maintained as a secured area, restricted only to authorized personnel. Locked cabinets or refrigerators/freezers are provided for secure storage of samples, subsamples and/or sample extracts. Laboratory personnel will take all the necessary actions to ensure that the designated holding areas for long term storage of COC samples are contamination free. Holding areas will be exclusively designated only for COC use and not be located in close proximity to laboratory chemicals. COC samples suspected of containing high concentrations of a contaminating substance will be sealed in a prescribed manner or kept in a different holding area to prevent sample-to-sample cross contamination.

Disposal of a physical sample held for litigation purposes shall occur only with the official written concurrence of the responsible legal representative, client, data user, sample submitter or other designated authority. All conditions of disposal and all correspondence between parties concerning the final disposition of the physical sample shall be recorded and retained as part of the COC documentation. Records shall indicate the date of disposal and the manner in which the disposal takes place. This could include, but is not limited to commentary that the sample was depleted during analysis, the sample was sent to a hazardous waste processing facility with appropriate confirming documentation or the sample was returned to the client. The COC document should terminate with the disposal phase clearly showing the signature of the individual performing this task. If the client or other assigned individual is receiving the sample for final disposal, this should also be clearly reflected on the COC document. The analytical report and all components of the Chain of Custody documentation will be retained as a permanent laboratory record. It will be filed and referenced by laboratory accession code and made available as a public document to all interested parties via the protocols of the Freedom of Information Act.

VII. Collection Procedures for Specific Types of Samples

A. Public Drinking Water

When drinking water is collected for testing, the site of collection must best represent the most frequent source of water used for human consumption within a residence. This is generally a kitchen or bathroom sink. Avoid the use of outdoor taps as collection points for drinking water samples. Sink fixtures used as a sample collection source must have supplemental plumbing hardware such as aerators, filters, strainers, splash guards, hoses and purification devices removed prior to sampling. If chemical water softeners, ion exchangers, charcoal filters or other large high-volume water-treatment devices are incorporated into a plumbing system, sample collectors shall consider the impact of this on the "representativeness" of the sample and disconnect or by-pass these devices in the most appropriate manner. Unless otherwise instructed, water from taps shall be run to waste for a minimum of two minutes prior to sample collection. The flow of water should be adjusted to minimize splashing or excessive turbulence during sampling. Containers shall not be rinsed or overfilled with sample unless there are specific instructions to conduct the sampling in this manner. During the actual collection process, the lip of the sample container must not come in physical contact with the faucet. The amounts of preserving agents which have been added to containers at the laboratory are appropriate for the volume of sample that container will hold. Special care must be exercised when collecting

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samples for bacteriological examination. Since coliform organisms are a normal component of the human intestinal tract and tend to be ubiquitous in the environment, they can easily be introduced as a contaminant during sampling activities. Inadvertent contamination by improper handling can easily lead to false positive findings. Some simple precautionary measures include the following:

- 1. Wash hands with soap and water prior to handling containers and collecting samples. Disposable wipes moistened with rubbing alcohol way also be used.
- 2. Avoid sampling from taps that are leaking or where the external surfaces of the tap is visibly moist. Wet surfaces promote the formation of biofilms where there is an ideal environment for bacteria to thrive.
- 3. Keep containers closed until just prior to sampling and close them immediately after sampling. Sampling containers should remain open just long enough to collect the sample. Covers must be replaced at quickly as possible following collection.
- 4. Hold the container in one hand and its cover in the other hand during the collection process. Do not set container covers down on counter tops, shelves or any other nearby surface.
- 5. Always use aseptic handling techniques. Handle the container and its cover by their outside surfaces paying careful attention not to touch the inside of the cover or any of the threaded surfaces on the lip of the container.

B. Surface Water and Wastewater

Water other than drinking water can originate from a wide variety of sources that include pipe discharges, ponds, puddles, storm water run-off, recharge basins, lakes, streams, marshes, wetlands, estuaries, monitoring wells, swimming pools, beaches, etc. Clean ladles, dippers, bailers, aspiration pipets or similar devices may be used to transfer water into the proper type of collection container. Splashing or excessive turbulence shall be avoided during sample collection. Containers shall not be rinsed or overfilled with sample unless there are specific instructions to conduct the sampling in this manner. The amounts of preserving agents which have been added to containers at the laboratory are appropriate for the volume of sample that container will hold.

C. Residues from Surfaces

Dust and dirt deposited on smooth surfaces such as floors, window sills, window wells and walls can be tested for the presence of environmental contaminants such as lead and PCBs using a wipe procedure. This technique does not apply to heavy textured surfaces such as rugs, upholstery, stucco, etc. Wipe samples should include detailed notes that defines the sampling location, surface type, surface composition, the physical dimensions of the surface under evaluation and its abatement status as well as any relevant historical perspectives that may be significant.

Using a plastic template or tape measure an area one foot by one foot shall be marked off at each sampling site. Some sampling areas may be irregular and the exact physical dimensions must be properly recorded. This is important since results are reported and interpreted in terms of the total mass of a measured contaminant per unit area of surface.

Pre-packaged, non-alcoholic, baby wipes composed of paper shall be provided by the laboratory for wipe testing. Baby wipes are ideally suited for this purpose because they are well sealed, the level of

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background contamination can be periodically monitored and they contain just the right amount of moisture that is necessary for minimizing smearing and streaking problems. Analytical grade filter paper moistened with a small amount of distilled water may be substituted if required. Unfold the paper and place it flat on the pre-marked surface at one of the corners. Gently rub (DO NOT SCRUB) the paper in a "S-LIKE" back-and-forth motion until the entire marked surface has been wiped one time. Fold the paper wipe in half with the dirty side folded to the inside. Rub the marked surface a second time starting in an adjacent corner so the "S-LIKE" motion will be perpendicular to the first wiping motion. Continue the back-and-forth pattern until the entire surface has been completely wiped a second time. Fold the paper in half a second time so that the dirty side is again toward the inside of the fold. For inorganic analytes (such as lead) place the folded paper in a 50 ml disposable plastic centrifuge tube or 3" by 6" plastic zip-lock bag. For organic analytes (such as PCBs or pesticides) place the folded paper in a clean glass vial with a Teflon lined cap. Labeling on the centrifuge tube or bag shall contain all pertinent sampling information to make identification clear and unambiguous. If the sampled surface contains an unusually heavy accumulation of dust or residue, it will be necessary to repeat the wipe procedure with a second paper wipe. The second wipe shall be treated as a separate sample and labeled accordingly. Wiping a smaller area is another alternative when dealing with a heavy accumulation of surface residue.

D. Paint Chips

At the time of this revision, paint chips are exclusively tested for the presence of lead, but could easily be tested for other analytes if the need arises. Collect with the aid of forceps several loose chips of paint from a peeling surface which are at a height that is readily accessible to small children. Collect enough paint chips such that the sample represents approximately two square inches of painted surface. If loose chips are not readily available, use a clean scraping tool to gently pry away a sufficient quantity of paint fragments.

Avoid excessive pressure when using a scraping tool during the collection process. This will reduce the possibility of contamination by plaster, fiber board, wood particles or other extraneous material. Place each sample in a LP4A (NYS Protocol) sample envelope, a 3" by 6" plastic zip-lock bag, an equivalent envelope/bag provided by the laboratory specifically for the collection of paint chips or a 50 ml. disposable plastic centrifuge tube. Transport at ambient temperature to the laboratory for analysis. Record all appropriate collection information on the envelope/bag/centrifuge tube so that identification is clear and unambiguous. Samples should not be combined. A separate envelope, bag or centrifuge tube must be used for each paint chip sample. A lead hazard is indicated if the paint chip contains more than 0.5% lead by weight.

E. Solder

The testing of solder for lead content was implemented to address the Nassau County Department of Health's regulations requiring that all new plumbing fixtures be installed using solder the contains less than 0.5% lead. Solder used prior to these regulations varied in lead content between 40% and 50%.

Solder should be collected from plumbing fixtures at a pipe junction in close proximity to the kitchen sink. Using a hardened steel knife blade, scrape off a solder fragment that is approximately one square centimeter in area. This should provide close to the 0.2 grams of material necessary for an analysis. Heavy solder accumulation in the form of hardened droplets will make an ideal sample. Position the edge of the knife blade flat against the pipe surface at the collection point and carefully

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scrape the soldered area with a motion that is parallel to the pipe surface. Deep penetrations with the knife blade that cut into copper pipe below the solder layer will include copper shavings that produce erroneous results. Collect the shavings in a clean 250 ml polyethylene or polypropylene jar with a plastic cap or 50 ml. disposable, plastic centrifuge tube and transport unpreserved to the laboratory at ambient temperature.

F. Soil

Soil samples shall be collected in such a manner so as to be representative of a suspected zone of contamination. Choose an area devoid of vegetation. If such an area is not readily available, trim the vegetation down to approximately 1/4 inch above the soil surface without disturbing or loosening the soil. One soil sample shall be a composite of five soil sections taken within a one square meter area. Using a tape measure, roughly mark off the one square meter area selected as the sampling site. Four of the sample sections shall be taken near the corners of the marked area and one in the center. Use a clean stainless steel trowel, spatula or similar tool to scoop out a section of soil approximately 2" x 2" x 2" in volume at each of the five locations and combine them together in a clean polyethylene or glass jar depending on the analytes under investigation. Use the compatible cap or closure provided with the container and transport the sample unpreserved back to the laboratory for analysis at ambient temperature unless instructed otherwise. A clean collection tool shall be used at each new sampling location to prevent cross contamination. Avoid including large rocks, roots or other debris in the sample which may not be truly representative of the soil under investigation.

VIII. Preservatives

The safe handling of all chemical agents necessary for the preservation of environmental samples is an important aspect of the sample collection process that may adversely impact on the well-being of he sample collector and any residents of the community who may be nearby while samples are being collected. Most preservatives are added to empty containers in the laboratory prior to sampling collection to minimize the risk of field contamination, but sample collectors should be astutely aware of the presence of these preservatives and the potential hazards that may be associated with them. A limited number of preservatives are also provided in solution form and added to the sample by the collector in the field at the time of collection. It is important that these hazardous chemical solutions be properly stored and handled to prevent spillage.

The more common solid preservatives such as sodium thiosulfate or ascorbic acid do not represent any appreciable hazard. Liquid preservatives that are strongly acidic or alkaline require special caution and care during handling. The 50% solution of hydrochloric acid used for preserving organic analytes is strong enough to cause minor chemical burns especially if it is unknowingly left on the skin for any extended period of time. Concentrated Nitric Acid is used in the preservation of metallic analytes and represents a serious hazard. If a sample container has been pretreated with nitric acid preservative prior to sampling, the sample bottle must be remain tightly capped until just prior to the moment of collection. Nitric acid is a strong oxidizer as well as a strong acid and quickly causes severe chemical burns. The first sign of a nitric acid burn is a yellow discoloration of the skin well before any noticeable burning sensation. At the first sign of this yellow discoloration, the affected area must be rinsed thoroughly with large volumes of running water as quickly as possible. Nitric acid will impart this same kind of yellow stain to counter tops and other surfaces if spillage occurs. Staining of surfaces can be prevented if the acid is promptly rinsed away, but once a noticeable yellow coloration has developed, the stain is likely to be permanent. An additional concern is that both the hydrochloric

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and nitric acid preservatives can release a significant amount of chemical fumes during the collection process that are caustic and can cause significant respiratory distress if inhaled. Sample collectors are advised to maintain a safe breathing distance from the open containers with such preservatives at the time of sampling. The alkaline reagent used for the preservation of dissolved oxygen by the Winkler Method or Cyanide is very caustic. It is first detected as an itching or tingling sensation and/or a slippery feeling on the skin. If not immediately rinsed off with large volumes of running water, it may result in blistering and the formation of scar tissue.

IX. Method Defined Analytes

Several environmental analytes are not true chemical or biological entities in their own right, but are method defined hybrids of something that has been pseudo-defined as a measurable environmental analyte. Because they are method defined, greater care must be taken in making meaningful interpretations regarding assessment and remedial measures needed to correctly address an environmental problem. Users of this type of analytical data must understand how the method defines these analytes, how method definitions may differ and how these differences may create interpretive ambiguities. It is particularly important in these cases to recognize what kind of analytical data is legitimately comparable and which is not. If uniformity of methodology is not properly recognized as a significant variable, then faulty interpretations may inevitably result.

A. Indicator Bacteria

A wide variety of pathogenic bacteria are capable of surviving in water. Throughout history, mankind has been victimized by cholera, typhoid, leptospirosis, schistosomiasis, chronic dysentery and a host of other diseases where water is cited as the primary vector of transmission. The consumption of contaminated water is, without reservation, the primary cause of numerous, large-scale disease epidemics. Waterborne outbreaks of disease continue to be a major public health problem since pathogenic bacteria such as Salmonella, Shigella, Campylobacter, Vibrio cholerae, Leptospira, Yersina and several virulent strains of Escherichia coli are readily transmitted by water.

There are literally thousands of different strains of bacteria that are ubiquitous in the environment. Most are soil organisms associated with the natural decay of plant material and have no sanitary significance. Efforts to arbitrarily identify the possible strains of bacteria in an environmental sample would be scientifically intense and have little public health value. The routine examination of water for specific bacterial pathogens is also impractical. This type of testing is extremely expensive, highly complex, requires sophisticated laboratory facilities and requires highly trained personnel. In addition, the availability of sufficiently sensitive laboratory procedures for identifying bacterial pathogens in the environment are very limited or non-existent. The need to find a simpler way to "indicate" their potential presence became a crucial public health objective. Since the feces and urine of warm-blooded animals is the single most significant source of waterborne pathogens affecting humans, it was concluded that a non-pathogenic bacteria typically present in the excreta of warm-blooded animals could serve as this "indicator".

The concept of using "indicator bacteria" to demonstrate water quality dates back to as early as 1880. Since then, several different bacteria and/or groups of bacteria have been used as "indicators", but the most widely accepted has been the coliform group. The correlation between coliforms and the actual presence of known pathogens tends to be highly irregular, but since it is a very reliable link to fecal contamination, it provides strong indirect evidence suggesting the presence of pathogens. Water

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quality standards were then developed based on a presumed "indicator-pathogen" association rather than a direct "indicator-pathogen" relationship.

Indicator bacteria may become stressed or injured in a water environment and are subject to structural or metabolic damage. These injured bacteria are incapable of normal growth and colony formation under standard conditions. As a result, a substantial number of indicator bacteria that may initially be present in an environmental sample may not be detected. The number of stressed or injured bacteria in any given environmental sample depend on several factors which include the presence of toxic substances, optimal salinity, temperature extremes, pH extremes, prolonged holding times and/or exposure to solar radiation. Collectively, these factors can cause a significant underestimation of the indicator bacteria population and even false negative findings. Either of these occurrences will ultimately lead to an incorrect assessment of a potential public health hazard. To complicate matters even further, numerous studies tend to support the finding that pathogens are less susceptible than indicator bacteria to environmental stress and are better able to recover from their injuries than indicator bacteria. Proper collection and preservation procedures for bacteriological samples are crucial components of the testing effort. They are designed to both minimize environmental stress and promote optimal conditions for bacterial survival. Personnel engaged in sample collection must be aware of their important role in producing meaningful data.

1. Definition of Coliforms

The two analytical procedures used for detecting "coliform" bacteria in water each define the term "coliform" differently. The fermentation technique is commonly referenced as the "MPN" (Most Probable Number) or the "MTF" (Multiple Tube Fermentation) method. By MPN, a "coliform" is defined as all aerobic or facultative anaerobic, gram negative, non-spore-forming, rod shaped bacteria that ferment lactose with the production of gas and acid within 48 hoursat an incubation temperature of 35 degrees Celsius. Additional confirmatory steps complete this procedure. This method allows for the statistical enumeration of the coliform population which shall be reported as the number of bacteria present per 100 mls of sample.

The chromogenic technique known as "DST" (Defined Substrate Technology) is the technical basis for the "Colilert Presence/Absence Test". For the chromogenic technique, a "coliform" is defined as all bacteria possessing the enzyme beta-D-galactosidase and capable of cleaving a chromogenic substrate, resulting in the release of an indicator chromogen within an incubation period of 24 hours. The Colilert procedure distinguishes between total and E. coli by providing indicator substrates that respond to characteristic enzymes. The substrate O-nitrophenyl-beta-D-galactopyranoside (ONPG) changes from clear to yellow for total coliforms and 4-methylumbelliferyl-beta-D-glucuronide (MUG) exhibits fluorescence for E. coli. The Colilert procedure requires 24 hours for completion, and are reported as either "present" or "absent". The Colilert procedure does not provide a bacterial count.

In order to be consistent with the coliform definition, a test result is not official for final reporting purposes until after the required incubation period has elapsed. Experience has demonstrated that in most instances when samples are coliform positive, this presence is demonstrated by the required color change well before the 24 hour incubation period is over. As soon as a color change is observed, the result is officially a positive finding and may be immediately reported without waiting for the end of the prescribed incubation period. When the presence of total coliforms has been demonstrated in an environmental sample by any standard method, it has become a matter of procedural policy for the Division of Public Health Laboratories to attempt an identification of the contaminating organism(s) by genus and species. This has proven to be useful as a quality control practice in the laboratory and may also provide insight into potential sources of

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contamination, health effects and remedial actions that might be used to address the problem. This is particularly important when dealing with public drinking water supplies and distribution systems. The following table lists the specific bacteria species that are detectable as total coliforms. The most common ones are highlighted in bold faced type and occur with the following relative frequency:

| Enterobacter cloacae | 43 |
|--------------------------|----|
| | % |
| Enterobacter agglomerans | 9% |
| Serratia marcescens | 8% |
| Citobacter freundi | 8% |
| Klebsiella oxytoca | 7% |
| Klebsiella pneumoniae | 6% |
| Enterobacter sakazakii | 6% |

Enterobacter cloacae is the most frequently encountered total coliform species and is highly resistant to the effects of chlorine. It is also a common soil organism and present in the fecal material of most wild animals. Although not generally classified as major health hazards, many of these are known to act as opportunistic pathogens.

Summary of bacteria that meet the definition of a "coliform organism"

(Boldface type indicates the most frequently detected "coliform organisms")

| Genus | Species | Genus | Species |
|--------------|---------------------------------|-------------|------------------------|
| Genus | Species | Gerus | Species |
| Budvicia | B. aquatica | Klebsiella | K. ornithinolytica |
| | | | K. oxytoca |
| Buttiauxella | B. agrestis | | K. ozaenae |
| | | | K. planticola |
| Cedecca | C. davisae | | K. pneumoniae |
| | C. lapagel | | K. rhinoscleromatis |
| | C. neteri | | K. terrigena |
| | | | |
| Citrobacter | C. amalonaticus | Kluyvera | K. ascorbata |
| | C. diversus | | K. cryocrescens |
| | C. freundi | | |
| | | Leciercia | L. adecarboxylata |
| Enterobacter | E. aerogenes | | |
| | E. agglomerans | Moellerella | M. wisconsensis |
| | E. amnigenus | | |
| | E. asburiae | Providencia | P. rettgeri |
| | E. cloacae | | P. stuartii |
| | E. gergoviae | | |
| | E. intermedium | Rahnella | R. aquatilis |
| | E. sakazakii | | |
| | E. taylorae | Salmonella | S. arizona (3a strain) |
| | | | S. arizona (3b strain) |
| Erwinia | E. cartovora (var. atroseptica) | | |
| | E. cartovora (var. cartovora) | Serratia | S. ficaria |

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| | E. herbicola (var. anamus) | | S. fonticola |
|-------------|-------------------------------|----------|-------------------|
| | E. herbicola (var. herbicola) | | S. liquefaciens |
| | E. pectobacterium | | S. marcescens |
| | E. rhapontici | | S. odorifera |
| | E. stewartii | | S. plymuthica |
| | E. uredovora | | S. rubidaea |
| | | | |
| Escherichia | E. coli (var. acidilactic) | Shigella | S. flexneri |
| | E. coli (var. communior) | | S. sonnei |
| | E. fergusonii | | |
| | E. freundi | Yersinia | Y. bercovieri |
| | E. hermannii | | Y. entericolitica |
| | E. vulneris | | Y. frederiksenii |
| | | | Y. intermedia |
| Hafnia | H. alvei | | |
| _ | | | Y. krisensenii |
| | | | Y. mollaretii |

2. Definition of Fecal Streptococcus

The fecal streptococcus group is defined as a group of bacteria capable of presumptive growth in dextrose broth containing 0.02% sodium azide within 48 hours of incubation at 35° Celsius and confirmatory growth on Pfizer Selective Enterococcus (PSE) Agar within 24 hours of incubation at 35° Celsius by the display of characteristic brownish-black colonies with brown halos. This group consists of several species of bacteria in the genus Streptococcus including Streptococcus faecalis, Streptococcus faecium, Streptococcus gallinarum, Streptococcus avium, Streptococcus bovis and Streptococcus equinus. Enumeration is accomplished by performing the presumptive phase of the test using a multiple tube serial dilution format that allows for the statistical calculation of a "Most Probable Number" (MPN). Results are reported as the number of bacteria present per 100 mls of sample.

Historically, fecal streptococcus and fecal coliform counts have been used to differentiate between human fecal contamination and fecal contamination derived from other warm-blooded animals. Fecal coliform to fecal streptococcus ratios (FC/FS) of 4.0 or higher supposedly indicates the predominance of human waste while ratios of 0.6 or lower indicate the predominance of waste from other warm-blooded animals. Recent studies have shown that the survival rates of the different Streptococcus species are highly variable in different aquatic environments and may be impacted disproportionately by other factors such as temperature, salinity and the residual presence of disinfectants. This upsets the legitimacy of the ratio and produces misleading results. THE USE OF THE FC/FS RATIO AS A MEANS OF DIFFERENTIATING BETWEEN HUMAN AND ANIMAL SOURCES OF POLLUTION IS NOT RECOMMENDED.

3. Definition of Enterococcus

The enterococcus group of bacteria is a subgroup of the fecal streptococci consisting of the species *Enterococcus faecalis, Enterococcus faecium, Streptococcus gallinarum*, and *Streptococcus savium*. It is related to the group "Fecal Streptococcus" in much the same way that "Fecal Coliforms" are related to "Total Coliforms". The Enterococcus Group of bacteria possess the enzyme enterococcus beta-glucosidase which is capable of activating the chromogen indoxyl-beta-

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D-glucoside whose characteristic blue color allows for the identification of enterococci. Using the selective culture media known as "mEI" Agar (Membrane-Enterococcus Indoxy-beta-D-Glucoside) the US Environmental Protection Agency has developed a single step procedure (EPA Method 1600) for enumerating enterococci. This method allows for the direct count of bacteria based on the development of colonies on the surface of a membrane filter. Following filtration, the bacterial cells on the membrane surface are place in contact with mEI agar and incubated at 41 degrees Celsius for 24 hours. All colonies with a blue halo are recorded as enterococci regardless of a colony's primary coloration.

Studies conducted by the U.S. Environmental Protection Agency indicate that the enterococcus group is the most efficient bacterial indicator for determining the extent of fecal contamination of recreational surface waters for bathing beaches in both fresh and marine water environments. National water quality guidelines using the enterococcus group as the biological indicator of choice has been established as the successor to the "Total Coliform" and "Fecal Coliform" indicators. The following reasons have been cited for using the enterococcus group as a more accurate indicator for fecal pollution.

- a. Enterococcus bacteria are present in smaller numbers than coliform bacteria, therefore it is easier to establish more meaningful standards or guidelines with narrower ranges of acceptance.
- b. Enterococcus bacteria are less able to survive in water or soil for extended periods of time, therefore their presence is a clearer indication of recent fecal contamination. The presence of coliform bacteria alone indicates fecal contamination at a much earlier time.
- c. The number of enterococcus bacteria tends to drop significantly as the distance from a pollution source increases. This facilitates the ability to pin-point sources of fecal contamination with greater accuracy. Since coliform bacteria tend to persist for longer periods of time and at great distances from the source, the use of coliform counts to determine the location of contamination source is significantly more difficult.
- d. Enterococcus bacteria cannot multiply outside of the intestinal tract of their human or animal host and are therefore more representative of the extent of fecal contamination.

 Coliform group bacteria are capable of reproducing in the environment.
- e. Enterococcus bacteria are not found in pure water, virgin soils or sites that are clearly isolated from human or animal life. They are only associated with a genuine fecal source. Many of the coliform group bacteria are non-fecal strains and are naturally occurring in oil. Their presence may not necessarily be associated with a fecal source and can cause interpretation difficulties.
- f. The number of enterococcus bacteria correlate better with number of bathers than does the total coliform or fecal coliform count.
- g. Enterococcus bacteria are capable of surviving at slightly higher residual chlorine levels than coliform group bacteria, producing a clearer indication of chlorination effectiveness.
- h. There is a much better relationship between the number of enterococcus bacteria and the incidence of swimming-related gastroenteritis than there is for the number of coliforms.

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i. There is close agreement between the decision-making results generated by the use of coliform and enterococcus test results in demonstrating adherence to a numerical water quality standard. Enterococcus data tends to generally be more sensitive and/or less specific and can be expected to cause more excedances of the accepted standard. A small increase in the number of beach closings per season can be expected when the enterococcal standard is adopted.

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IX. Summary of Specific Sampling and Handling Requirements

Category I - Environmental Analyses / Potable Water Subcategory A - Drinking Water Non-Metals (One liter is sufficient for all analytes in this Subcategory).

| | | Minimum | | | T |
|-------------------------|-----------------|-------------|----------------------|---------------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |
| Analyte | Container | volume (mi) | Tilling Requirements | Treservation | Wax Holding Time |
| Alkalinity | Polyethylene or | 500 | Fill Completely Full | Cool to 4 C | 14 Days |
| · | Polypropylene | | with No Trapped Air | | , |
| Calcium Hardness | Polyethylene or | 100 | None | Adjust with | 6 Months |
| | Polypropylene | | | concentrated Nitric | |
| | | | | acid to pH of <2. | |
| Chloride | Polyethylene or | 50 | None | None | 28 Days |
| | Polypropylene | | | | |
| Color | Polyethylene or | 100 | None | Cool to 4 C | 48 Hours |
| | Polypropylene | | | | |
| Corrosivity | Polyethylene or | 500 | Fill Completely Full | Cool to 4 C | 1 Hour |
| | Polypropylene | | with No Trapped Air | | |
| Fluoride | Polyethylene or | 50 | None | None | 28 Days |
| | Polypropylene | | | | |
| Nitrite-N | Polyethylene or | 50 | None | Cool to 4 C | 48 Hours |
| | Polypropylene | | | | |
| Nitrate-N | Polyethylene or | 50 | None | Cool to 4 C | 48 Hours |
| | Polypropylene | | | | |
| Hydrogen Ion (pH) | Polyethylene or | 100 | None | None | 1 Hour |
| | Polypropylene | | | | |
| Orthophosphate | Polyethylene or | 50 | None | Cool to 4 C | 48 Hours |
| | Polypropylene | | | | |
| Solids, Total Dissolved | Polyethylene or | 200 | None | Cool to 4 C | 7 Days |
| | Polypropylene | | | | |
| Specific Conductance | Polyethylene or | 200 | None | Cool to 4 C | 28 Days |
| | Polypropylene | | | | |
| Silica Dissolved | Polyethylene or | 50 | None | Cool to 4 C | 28 Days |
| | Polypropylene | | | | |
| Sulfate | Polyethylene or | 50 | None | Cool to 4 C | 28 Days |
| | Polypropylene | | | | |
| Turbidity | Polyethylene or | 50 | None | Cool to 4 C | 48 Hours |
| | Polypropylene | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory B - Drinking Water Miscellaneous (one liter of sample is sufficient for all analytes)

| Di (2-ethylhexyl)- adipate | Amber glass with | 1000 | None | Cool to 4 C. Add | Extract within 14 |
|-------------------------------|-----------------------|------|------|---------------------|--------------------|
| Bis (2-ethylhexyl)- phthalate | TFE lined cap. | | | 60mg of Sodium | days of sample |
| Benzo(a)pyrene | Sampling equipment | | | Thiosulfate to | collection. |
| Butachlor | must be free of | | | remove any chlorine | Complete analysis |
| Hexachlorobenzene | plastic tubing, | | | residual. Adjust pH | within 30 days of |
| Hexachlorocyclopentadiene | gaskets or other | | | to <2 with 6N HCL. | sample extraction. |
| Propachlor | plastic surfaces that | | | Protect from | |
| Тюристог | may leach | | | exposure to light | |
| | contaminants into the | | | | |
| | sample. | | | | |

Subcategory C - Volatile Halocarbons

| Bromochloromethane | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. Add | 14 Days |
|---------------------------|--------------------|-----------------|-----------------------|------------------------|---------|
| Bromomethane | borosilicate glass | | with no entrapped air | ascorbic acid at | |
| Carbon Tetrachloride | vials with Teflon | | bubbles. Minimize | 25mg/40ml (Added | |
| Chloroethane | lined screw caps. | | agitation during the | to empty vial prior to | |
| Chloromethane | | | collection process. | collection). Adjust | |
| Dibromomethane | | | | pH with 1:1 HCL to a | |
| Dichlorodifluoromethane | | | | pH of <2. | |
| 1,1-Dichloroethane | | | | | |
| 1,2-Dichloroethane | | | | | |
| Cis-1,2-Dichloroethene | | | | | |
| Trans-1,2-Dichloroethene | | | | | |
| 1,2-Dichloropropane | | | | | |
| 1,3-Dichloropropane | | | | | |
| 2,2-Dichloropropane | | | | | |
| 1,1-Dichloropropene | | | | | |
| Cis-1,3-Dichloropropene | | | | | |
| Methylene chloride | | | | | |
| 1,1,1,2-Tetrachloroethane | | | | | |
| 1,1,2,2-Tetrachloroethane | | | | | |
| Tetrachloroethene | | | | | |
| 1,1,1-Trichloroethane | | | | | |
| Trichloroethene | | | | | |
| 1,1,2-Trichloropropane | | | | | |
| Trichlorofluoromethane | | | | | |
| 1,2,3-Trichloropropane | | | | | |
| Vinyl chloride | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory D - Drinking Water Chlorinated Acids (One liter of sample is sufficient for all analytes)

| 2,4-D | Amber borosilicate | 1000 | None | Cool to ≤6 C. Add | Extract within 14 |
|-------------------|----------------------|------|------|---------------------|----------------------|
| Dicamba | glass with TFE lined | | | 60 mg of Sodium | days of sample |
| Dinoseb | cap. | | | Thiosulfate to | collection. |
| Pentachlorophenol | | | | remove any chlorine | Complete analysis |
| Pichloram | | | | residual. Protect | within 14 days after |
| 2,4,5-TP (Silvex) | | | | from exposure to | extraction. |
| , ,- () | | | | light. | |

Subcategory E - Polychlorinated Biphenyls (One liter is sufficient for all analytes in this Subcategory)

| PCB 1016 | Amber borosilicate | 1000 | None | Cool to ≤6 C. Add | Extract within 14 |
|----------|----------------------|------|------|---------------------|----------------------|
| PCB 1221 | glass with TFE lined | | | 60 mg of Sodium | days of sample |
| PCB 1232 | cap. | | | Thiosulfate to | collection. |
| PCB 1242 | | | | remove any chlorine | Complete analysis |
| PCB 1248 | | | | residual. Protect | within 14 days after |
| PCB 1254 | | | | from exposure to | extraction. |
| PCB 1260 | | | | light. | |

Subcategory F - Drinking Water Bacteriology

| Coliform, Total | Plastic IDEXX | 100 | Fill to marked fill line | Cool to 4 C. Sodium | 30 Hours |
|----------------------|------------------------|-----|--------------------------|---------------------|----------|
| Coliform, E. Coli | vessel, pre-sterilized | | using sterline handling | Thiosulfate pre- | * |
| | with sodium | | technique | added at 0.008% | |
| | Thiosulfate already | | | | |
| | added (Colilert | | | | |
| | trademark) | | | | |
| Standard Plate Count | Plastic IDEXX | 10 | Use sterile handling | Cool to 4 C. Sodium | 8 Hours |
| | vessel, pre-sterilized | | technique at all times | Thiosulfate pre- | * |
| | with sodium | | | added at 0.008% | |
| | Thiosulfate already | | | | |
| | added (Colilert | | | | |
| | trademark) | | | | |

^{*} Maximum holding time includes the time elapsed from collection of the sample to placement into the incubator

Subcategory G - Drinking Water Trihalomethanes

| Bromodichloromethane | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. Add | 14 Days |
|----------------------|--------------------|-----------------|-----------------------|----------------------|---------|
| Bromoform | borosilicate glass | | with no entrapped air | ascorbic acid at | |
| Dibromochloromethane | vials with Teflon | | bubbles. Minimize | 25mg/40ml. Adjust | |
| Chloroform | lined screw caps. | | agitation during the | pH with 1:1 HCL to a | |
| | | | collection process. | pH of <2. | |

3-Hydroxycarbofuran

Methomyl Oxamyl Env. Sample Acceptance Policy Doc. No. L-6, Rev. No. 4 Date: March 31, 2010 Page 28 of 45

chloroacetic acid.

extraction.

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| | | Minimum | | | |
|-------------------------------------|-------------------------------|-------------------|----------------------------|---------------------------------|---------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |
| Subcategory H - Subcategory). | Drinking Water I | Metals I (A sa | ample volume of 250 n | nls. is sufficient for a | all analytes in thi |
| Arsenic, Total | Polyethylene or | 100 | None | Adjust with | 6 Months |
| Barium,Total | Polypropylene | | | concentrated Nitric | |
| Cadmium, Total | | | | acid to pH of <2. | |
| Chromium, Total | | | | | |
| Copper, Total | | | | | |
| ron, Total | | | | | |
| ead, Total | | | | | |
| Manganese, Total | | | | | |
| Mercury, Total | | | | | |
| Selenium, Total | | | | | |
| Silver, Total | | | | | |
| Sodium, Total | | | | | |
| Zinc, Total | | | | | |
| Antimony, Total Beryllium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with concentrated Nitric | 6 Months |
| | Polypropylene | lene | | acid to pH of <2. | |
| Nickel, Total Thallium, Total | | | | | |
| mamum, rotai | | | | | |
| Subcategory J - 1 | Microextractables | (One liter of sar | nple is sufficient for all | l analytes in this Sub | category). |
| ,2-Dibromomethane | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. | 28 Days. |
| 1,2-Dibromo-3 | borosilicate glass | | with no entrapped air | Add ascorbic acid at | |
| chloropropane | vials with Teflon | | bubbles. Minimize | 25mg/40ml. Adjust | |
| | lined screw caps. | | agitation during the | pH with 1:1 HCL wot | |
| | | | collection process. | a pH of <2. | |
| Subcategory K - | Drinking Water M | // Methylcarbam | nate Pesticides | | <u> </u> |
| suscuregory ri | 27 mining (, uver 1 | i com ji car san | | | |
| Aldicarb | 40 ml glass vial with | 40 ml. per vial | None | Cool to 4 C during | Extract within 14 |
| Aldicarb sulfone | PTFE faced silicone | TO IIII. poi viai | | transit and store at - | days of sample |
| Aldicarb sulfoxide | septa and screw cap | | | 10 C. Add 5 mg of | collection. |
| Carbaryl | | | | Sodium Thiosulfate | Complete analysis |
| Carbafuran | | | | and 1.8 ml of | within 28 days of |
| Carbofuran | | | | | extraction |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory L - Drinking Water Organohalide Pesticides (One liter is sufficient for all analytes)

| Alachlor Aldrin Atrazine Chlordane Dieldrin Endrin Heptachlor Heptachlor epoxide Lindane Methoxychlor Metolachlor Metribuzin Simazine Toxaphene | Amber borosilicate glass with TFE lined cap. Sampling equipment must be free of plastic tubing, gaskets or other plastic surfaces that may contaminate the sample. | 1000 | None | Cool to ≤6 C. Add 60 mg of Sodium Thiosulfate to remove any chlorine residual. Protect from exposure to light Adjust with 6N HCL to a pH of <2. Store refrigerated. | Extract within 14 days of sample collection. Complete analysis within 30 days after extraction. |
|---|--|------|------|---|---|
|---|--|------|------|---|---|

Subcategory M - Volatile Aromatics

| Benzene | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. Add | 14 Days |
|------------------------|--------------------|-----------------|-----------------------|-----------------------|---------|
| Bromobenzene | borosilicate glass | | with no entrapped air | ascorbic acid at 25 | - |
| n-Butylbenzene | vials with Teflon | | bubbles. Minimize | mg/40 ml (Added to | |
| Butylbenzene | lined screw caps. | | agitation during the | empty vial prior to | |
| tert-Butylbenzene | | | collection process. | collection) Adjust pH | |
| Chlorobenzene | | | | with 1:1 HCL to a pH | |
| 2-Chlorotoluene | | | | of <2. | |
| 4-Chlorotoluene | | | | | |
| 1,2-Dichlorobenzene | | | | | |
| 1,3-Dichlorobenzene | | | | | |
| 1,4-Dichlorobenzene | | | | | |
| Ethyl benzene | | | | | |
| Hexachlorobutadiene | | | | | |
| Isopropylbenzene | | | | | |
| p-Isopropyltoluene (p- | | | | | |
| Cumene) | | | | | |
| n-Propylbenzene | | | | | |
| Styrene | | | | | |
| Toluene | | | | | |
| 1,2,3-Trichlorobenzene | | | | | |
| 1,2,4-Trichlorobenzene | | | | | |
| 1,2,4-Trimethylbenzen | | | | | |
| 1,3,5-Trimethylbenzene | | | | | |
| m-Xylene | | | | | |
| o-Xylene | | | | | |
| p-Xylene | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

2.) Category II - Environmental Analyses / Non-Potable Water

Subcategory A - Chlorinated Hydrocarbon PesticidesNote: (One liter is sufficient for all analytes)

| 4,4'-DDD | Amber borosilicate | 1000 | None | Cool to 4 C. Add 60 | Extract within 14 |
|--------------------|-------------------------|------|------|----------------------|----------------------|
| 4,4'-DDE | glass with TFE lined | | | mgh of Sodium | days of sample |
| 4,4'-DDT | cap. Sampling | | | Thiosulfate to | collection. |
| alpha-BHC | equipment must be | | | remove any chlorine | Complete analysis |
| Aldrin | free of plastic tubing, | | | residual. Protect | within 30 days after |
| beta-BHC | gaskets or other | | | from exposure to | extraction. |
| Captan | plastic surfaces that | | | light Adjust with 6N | |
| Chlordane, Total | may contaminate the | | | HCL to a pH of <2. | |
| delta-BHC | sample. | | | Store refrigerated. | |
| Dichloram | | | | | |
| Dicofol | | | | | |
| Dieldrin | | | | | |
| Endrin aldehyde | | | | | |
| Endrin | | | | | |
| Endosulfan I | | | | | |
| Endosulfan II | | | | | |
| Endosulfan sulfate | | | | | |
| Heptachlor | | | | | |
| Heptachlor epoxide | | | | | |
| Isodrin | | | | | |
| Lindane | | | | | |
| Mirex | | | | | |
| Methoxychlor | | | | | |
| Perthane | | | | | |
| Trifluralin | | | | | |
| Toxaphene | | | | | |

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| Ī | | | Minimum | | | |
|---|---------|-----------|-------------|----------------------|--------------|------------------|
| | Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory B - Purgeable Halocarbons

| Bromodichloromethane | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. | 14 Days |
|----------------------------|--------------------|-----------------|-----------------------|------------------------|---------|
| Bromoform | borosilicate glass | | with no entrapped air | Add ascorbic acid at | |
| Bromomethane | vials with Teflon | | bubbles. Minimize | 25mg/40ml (Added | |
| Carbon Tetrachloride | lined screw caps. | | agitation during the | to empty vial prior to | |
| Chloroethane | | | collection process. | collection). Adjust | |
| Chloroform | | | | pH with 1:1 HCL to a | |
| Chloromethane | | | | pH of <2. | |
| Dibromochloromethane | | | | | |
| Dichlorodifluoromethane | | | | | |
| 1,1-Dichloroethane | | | | | |
| 1,2-Dichloroethane | | | | | |
| 1,1-Dichloroethene | | | | | |
| 1,2-Dichloroethene (total) | | | | | |
| 1,2-Dichloropropane | | | | | |
| cis-1,3-Dichloropropene | | | | | |
| trans-1,3-Dichloropropene | | | | | |
| Methylene chloride | | | | | |
| 1,1,2,2-Tetrachloroethane | | | | | |
| Tetrachloroethene | | | | | |
| 1,1,1-Trichloroethane | | | | | |
| 1,1,2-Trichloroethane | | | | | |
| Trichloroethene | | | | | |
| Trichlorofluoromethane | | | | | |
| Vinyl chloride | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory C - Wastewater Miscellaneous

Note: A total sample volume of 1 liter is sufficient for all analytes in this Subcategory.

| Boron | Polyethylene or Polypropylene. Contact with borosilicate glass surfaces will cause major contamination that will invalidate the sample. | 50 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
|----------------------|--|-----|------|--|------------|
| Color | Polyethylene or Polypropylene | 100 | None | Cool to 4 C. | 48 Hours |
| Hydrogen Ion (pH) | Polyethylene or Polypropylene | 100 | None | None | 15 Minutes |
| Specific Conductance | Polyethylene or Polypropylene | 200 | None | Cool to 4 C | 28 Days |
| Silica, Dissolved | Polyethylene or Polypropylene. Contact with borosilicate glass surfaces will cause major contamination that will invalidate the sample. | 50 | None | Cool to 4 C | 28 Days |

Subcategory D - Wastewater Metals III (A sample volume of 250 mls. is sufficient for all analytes)

| Colbalt, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
|-------------------|----------------------------------|-----|------|--|-----------|
| Molybdenum, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
| Thallium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months. |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory E - Wastewater Bacteriology

| Coliform, Total | Pre-sterilized Whirl- | 100 | None | Cool to 4 C. | 8 Hours |
|----------------------|-----------------------|-----|-----------------------|--------------------|---------|
| Coliform, E. Coli | pak sample bag with | | | Add 80 mg of | * |
| | a sodium Thiosulfate | | | Sodium Thiosulfate | |
| | pellet already added | | | to remove any | |
| | or pre-sterilized | | | chlorine residual. | |
| | empty plastic | | | Protect from | |
| | collection bag | | | exposure to light. | |
| Standard Plate Count | Same as for | 100 | Same as for Coliforms | Same as for | 8 Hours |
| | Coliforms (See | | (See above). | Coliforms (See | *,# |
| | above). | | | above). | |

- * Maximum holding time includes the time elapsed from collection of the sample to placement into the incubator.
- # E. coli samples enumerated for reporting to EPA under the LT2 rule may be tested when the 8 hour hold time is exceeded and within 30 hours from the time of collection to set-up, only when preservation is documented intact. All data generated outside the 8 hour hold time must be qualified as such in the report to the client. No samples older than 30 hours shall be tested

Subcategory F - Chlorinated Hydrocarbons: (One liter is sufficient for all analytes in this Subcategory)

| 2-Chloronaphthalene | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 7 |
|---------------------------|--------------------|------|------|--------------------|----------------------|
| Hexachlorobenzene | glass jar with TFE | | | Add 80 mg of | days of sample |
| Hexachlorobutadiene | lined cap. | | | Sodium Thiosulfate | collection. |
| Hexachloroethane | | | | to remove any | Complete analysis |
| Hexachlorocyclopentadiene | | | | chlorine residual. | within 40 days after |
| 1,2,4-Trichlorobenzene | | | | Protect from | extraction. |
| 1,=,: | | | | exposure to light. | |

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| Ī | | | Minimum | | | |
|---|---------|-----------|-------------|----------------------|--------------|------------------|
| | Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory G - Wastewater Metals II (A volume of 250 mls. is sufficient for all analytes)

| Aluminum, total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
|------------------|----------------------------------|-----|------|--|----------|
| Antimony, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
| Arsenic, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
| Beryllium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Mercury, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 14 Days |
| Selenium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Vanadium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Zinc, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |

Subcategory H - Polynuclear Aromatics (One liter is sufficient for all analytes in this subcategory)

| Acenaphthene | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 7 |
|------------------------|-------------------------|------|------|----------------------|----------------------|
| Acenaphthylene | glass with TFE lined | | | Add 80 mg of | days of sample |
| Anthracene | cap. Sampling | | | Sodium Thiosulfate | collection. |
| Benzo(a)anthracene | equipment must be | | | to remove any | Complete analysis |
| Benzo(a)pyrene | free of plastic tubing, | | | chlorine residual. | within 30 days after |
| Benzo(b)fluoranthene | gaskets or other | | | Protect from | extraction. |
| Benzo(g,h,i)perylene | plastic surfaces that | | | exposure to light by | |
| Benzo(k)fluoranthene | may contaminate the | | | storing in darkness. | |
| Chrysene | sample. | | | Keep refrigerated. | |
| Dibenzo(a,h)anthracene | | | | | |
| Fluoranthene | | | | | |
| Fluorene | | | | | |
| Indeno(1,2,3-cd)pyrene | | | | | |
| Naphthalene | | | | | |
| Phenanthrene | | | | | |
| Pyrene | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory I - Phthalate Esters (A sample volume of 1 liter is sufficient for all analytes in this Subcategory)

| Butyl benzyl phthalate | Amber borosilicate | 1000 | None | Cool to 4 C. Protect | Extract within 14 |
|-------------------------------------|-------------------------|------|------|----------------------|----------------------|
| Bis (2-ethylhexyl) phthalate | glass with TFE lined | | | from exposure to | days of sample |
| Diethyl phthalate | cap. Sampling | | | light. Store | collection. |
| Dimethyl phthalate | equipment must be | | | refrigerated | Complete analysis |
| Di-n-butyl phthalate | free of plastic tubing, | | | | within 14 days after |
| Sulfate | gaskets or other | | | | extraction. |
| Di-n-octyl phthalate | plastic surfaces that | | | | |
| · · · · · · · · · · · · · · · · · · | may contaminate the | | | | |
| | sample. | | | | |

Subcategory J - Purgeable Aromatics

| Benzene | Duplicate 40 ml | 40 ml. per vial | Fill completely full | Cool to 4 C. Add | 14 Days |
|---------------------|--------------------|-----------------|-----------------------|------------------------|---------|
| Chlorobenzene | borosilicate glass | | with no entrapped air | ascorbic acid at | |
| 1,2-Dichlorobenzene | vials with Teflon | | bubbles. Minimize | 25/mg/40ml. (Added | |
| 1,3-Dichlorobenzene | lined screw caps. | | agitation during the | to empty vial prior to | |
| 1,4-Dichlorobenzene | | | collection process | collection). Adjust | |
| Ethylbenzene | | | | pH with 1:1 HCL to a | |
| Toluene | | | | pH of <2. | |
| Xylenes, Total | | | | | |

Subcategory K - Nutrients (A total volume of 1 liter is sufficient for all analytes in this subcategory)

| Ammonia-N | Polyethylene or Polypropylene | 50 | None | Cool to 4 C. | 28 Days |
|-------------------|----------------------------------|-----|------|--|----------|
| Nitrite-N | Polyethylene or Polypropylene | 50 | None | Cool to 4 C. | 48 Hours |
| Nitrate-N | Polyethylene or Polypropylene | 50 | None | Cool to 4 C. | 48 Hours |
| Orthophosphate | Polyethylene or Polypropylene | 50 | None | Cool to 4 C. | 48 Hours |
| Phosphorus, Total | Polyethylene or Polypropylene | 200 | None | Cool to 4 C. Adjust with sulfuric acid to a pH of <2 | 28 Days |

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| Ī | | | Minimum | | | |
|---|---------|-----------|-------------|----------------------|--------------|------------------|
| | Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory L - Triazine and O-Aryl Carbamate Pesticides

| Atrazine | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 14 |
|----------|-------------------------|------|------|--------------------------|----------------------|
| | glass with TFE lined | | | Add 80 mg of | days of sample |
| | cap. Sampling | | | Sodium Thiosulfate | collection. |
| | equipment must be | | | to remove any | Complete analysis |
| | free of plastic tubing, | | | chlorine residual. | within 14 days after |
| | gaskets or other | | | Protect from | extraction. |
| | plastic surfaces that | | | exposure to light. | |
| | may leach the | | | Adjust with 6N HCL | |
| | analytes into the | | | to a pH of < 2 . Store | |
| | sample as | | | refrigerated | |
| | contamination. | | | | |

Subcategory M - Demand

| Biochemical | Borosilicate glass | 300 | Fill to overflowing and | Adjust with 2 ml. of | 6 Hours (take first |
|-------------|--------------------|-----|--------------------------|-----------------------|---------------------|
| Oxygen | BOD bottle with | | add preservatives. | manganous sulfate | DO reading on the |
| Demand | narrow flared neck | | Avoid excess | reagent and 3 ml. of | day the sample is |
| | and tapered ground | | turbulence during the | alkaline iodide | collected). |
| | glass stopper. | | collection process. | reagent. Maintain the | |
| | | | Careful insertion of the | sample at the | |
| | | | ground glass stopper | temperature of | |
| | | | will prevent entrapped | collection. | |
| | | | air bubbles. Mix by | | |
| | | | gently inverting the | | |
| | | | sample bottle several | | |
| | | | times. | | |

Subcategory N - Chlorophenoxy Acid Pesticides (One liter is sufficient for all analytes)

| Dicamba | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 14 |
|------------------|--------------------|------|------|--------------------|----------------------|
| | glass jar with TFE | | | Add 80 mg of | days of sample |
| 245 | lined cap. | | | Sodium Thiosulfate | collection. |
| 2,4-D | | | | to remove any | Complete analysis |
| | | | | chlorine residual. | within 14 days after |
| 2,4,5-T | | | | Protect from | extraction. |
| | | | | exposure to light. | |
| | - | | | Acidify with 1:1 | |
| 2,4,5-TP(Silvex) | | | | HCL to a pH of <2. | |
| | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

$Subcategory\ O\ -\ Wastewater\ Metals\ I\ (\mbox{A volume of 250 mls. is sufficient for all analytes)}$

| Barium, total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of | 6 Months |
|------------------|----------------------------------|-----|------|---|----------|
| Cadmium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of | 6 Months |
| Calcium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of <2 | 6 Months |
| Chromium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Copper, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Iron, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Lead, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Magnesium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Manganese, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of | 6 Months |
| Nickel, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Potassium, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Silver, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Zinc, Total | Polyethylene or Polypropylene | 100 | None | Adjust with conc. Nitric Acid to a pH of | 6 Months |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory P – Minerals (A sample volume of 1 liter is sufficient for all analytes in this Subcategory).

| Alkalinity, Total | Polyethylene or Polypropylene | 50 | Fill completely full with no entrapped air | Cool to 4 C. | 14 Days |
|-------------------|----------------------------------|----|--|--|----------|
| Chloride | Polyethylene or Polypropylene | 50 | None | None | 28 Days |
| Fluoride, Total | Polyethylene or Polypropylene | 50 | None | None | 28 Days |
| Hardness, Calcium | Polyethylene or Polypropylene | 50 | None | Adjust with conc. Nitric Acid to a pH of < 2 | 6 Months |
| Hardness, Total | Polyethylene or Polypropylene | 50 | None | Adjust with conc. Nitric Acid to a pH of | 6 Months |
| Sulfate | Polyethylene or Polypropylene | 50 | None | Cool to 4 C. | 28 Days. |

Subcategory Q - Polychlorinated Biphenyls (One liter is sufficient for all analytes in this Subcategory).

| PCB 1016 | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 14 |
|-------------------|--------------------|------|------|--------------------|----------------------|
| PCB 1221 | glass jar with TFE | | | Add 80 mg of | days of sample |
| PCB 1232 | lined cap | | | Sodium Thiosulfate | collection. |
| PCB 1242 | | | | to remove any | Complete analysis |
| PCB 1248 | | | | chlorine residual. | within 30 days after |
| PCB 1254 | | | | Protect from | extraction. |
| PCB 1260 | | | | exposure to light | |
| 4-Nitrophenol | | | | | |
| Pentachlorophenol | | | | | |

Subcategory R - Priority Pollutant Phenols (One liter is sufficient for all analytes in this Subcategory).

| 4-Chloro-3-methyl phenol | Amber borosilicate | 1000 | None | Cool to 4 C. | Extract within 14 |
|----------------------------|--------------------|------|------|--------------------|----------------------|
| 2-Chlorophenol | glass jar with TFE | | | Add 80 mg of | days of sample |
| 2,4-Dichlorophenol | lined cap | | | Sodium Thiosulfate | collection. |
| 2,4-Dimethylphenol | | | | to remove any | Complete analysis |
| 2,4-Dinitrophenol | | | | chlorine residual. | within 14 days after |
| 2-Methyl-4,6-dinitrophenol | | | | Protect from | extraction. |
| 2-Nitrophenol | | | | exposure to light | |
| 4-Nitrophenol | | | | | |
| Pentachlorophenol | | | | | |
| Phenol | 1 | | | | |
| 2.4.6-Trichlorophenol | 1 | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory S - Residue (A total sample volume of 1 liter is sufficient for all analytes in this Subcategory).

| Solids, Total Dissolved | Polyethylene or | 200 | None | Cool to 4 C. | 7 Days |
|-------------------------|-----------------|-----|------|--------------|--------|
| | Polypropylene | | | | |
| Solids, Total Suspended | Polyethylene or | 200 | None | Cool to 4 C. | 7 Days |
| | Polypropylene | | | | |
| Solids, Total | Polyethylene or | 200 | None | Cool to 4 C. | 7 Days |
| | Polypropylene | | | | |

3.) Category IV - Environmental Analyses / Solid and Hazardous Waste Subcategory A - Metals II

| Antimony, Total | Polyethylene or | Variable | None | None | 6 Months |
|-----------------|-------------------|----------|------|------|----------|
| | Polypropylene Jar | | | | |
| Arsenic, Total | Polyethylene or | Variable | None | None | 6 Months |
| | Polypropylene Jar | | | | |
| Mercury, Total | Polyethylene or | Variable | None | None | 14 Days |
| | Polypropylene Jar | | | | |
| Selenium, Total | Polyethylene or | Variable | None | None | 6 Months |
| | Polypropylene Jar | | | | |

Subcategory A - Miscellaneous

| Lead in Dust Wipes | Analytical grade filter or pre-sealed baby wipe retained in a 50ml plastic centrifuge tube or plastic zip-lock bag | 1 Square Foot of surface for wiping (see special collection protocols | None | None | 6 Months |
|--------------------|---|--|------|------|----------|
| Lead in Paint | Paper Envelope | >100 mg (see special collection protocols) | None | None | 6 Months |

Subcategory B - Priority Pollutant Phenols

| 4-Chloro-3-methyl phenol | Amber borosilicate | Variable | None | Cool to 4 C. | Extract within 14 |
|----------------------------|--------------------|----------|------|--------------|----------------------|
| 2-Chlorophenol | glass jar with TFE | | | | days of sample |
| 2,4-Dichlorophenol | lined cap | | | | collection. |
| 2,4-Dimethylphenol | | | | | Complete analysis |
| 2,4-Dinitrophenol | | | | | within 14 days after |
| 2-Methyl-4,6-dinitrophenol | | | | | extraction. |
| 2-Nitrophenol | | | | | |
| 4-Nitrophenol | | | | | |
| Pentachlorophenol | | | | | |
| Phenol | | | | | |

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| 2,4,6-Trichlorophenol | | | |
|-----------------------|--|--|--|
| | | | |

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| | | EE/II Labout | 10337 | | | | | | | |
|--|--------------------------------------|--------------|----------------------|--------------------|----------------------|--|--|--|--|--|
| | | Minimum | | | | | | | | |
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time | | | | | |
| Subcategory C - Characteristic Testing | | | | | | | | | | |
| TCLP (Metals Only) | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Subcategory D - | Subcategory D - Metals I | | | | | | | | | |
| Barium, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Cadmium, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Chromium, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Lead, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Nickel, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Silver, Total | Polyethylene or Polypropylene Jar | Variable | None | None | 6 Months | | | | | |
| Subcategory 1 | E - Polychlorinate | ed Biphenyls | | | | | | | | |
| PCB 1016 | Amber borosilicate | Variable | None | Cool to 4 C. | Extract within 14 | | | | | |
| PCB 1221 | glass jar with TFE | | | Protect from | days of sample | | | | | |
| PCB 1231 | lined cap | | | exposure to light. | collection. | | | | | |
| PCB 1242 | | | | | Complete analysis | | | | | |
| PCB 1248 | | | | | within 30 days after | | | | | |
| PCB 1254 | | | | | extraction. | | | | | |
| PCB 1260 | | | | | | | | | | |
| Subcategory F - | Purgeable Aroma | atics | | | | | | | | |
| Benzene | Duplicate 40 ml | Variable | None | Cool to 4 C. | 14 Days | | | | | |
| Chlorobenzene | borosilicate glass | | | | | | | | | |
| 1,2-Dichlorobenzene | vials with Teflon | | | | | | | | | |
| 1,3-Dichlorobenzene | lined screw caps. | | | | | | | | | |
| 1,4-Dichlorobenzene | | | | | | | | | | |
| Ethylbenzene | | | | | | | | | | |
| Toluene | | | | | | | | | | |
| m-Xylene | | | | | | | | | | |
| o-Xylene | | | | | | | | | | |
| n Vylana | | | | | | | | | | |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory G - Chlorinated Hydrocarbon Pesticides

| Aldrin | Amber borosilicate | Variable | None | Cool to 4 C. Protect | Extract within 14 |
|---------------------|--------------------|----------|------|----------------------|----------------------|
| alpha-BHC | glass jar with TFE | | | from exposure to | days of sample |
| beta-BHC | lined cap | | | light | collection. |
| delta-BHC | | | | | Complete analysis |
| gamma-BHC (Lindane) | | | | | within 30 days after |
| Chlordane | | | | | extraction. |
| 4,4'-DDD | | | | | |
| 4,4'-DDE | | | | | |
| 4,4'-DDT | | | | | |
| Dieldrin | | | | | |
| Endosulfan I | | | | | |
| Endosulfan II | | | | | |
| Endosulfan sulfate | | | | | |
| Endrin | | | | | |
| Endrin aldehyde | | | | | |
| Heptachlor | | | | | |
| Heptachlor epoxide | | | | | |
| Methoxychlor | | | | | |
| Toxaphene | | | | | |

Subcategory H - Chlorinated Hydrocarbons

| 2-Chloronaphthalene | Amber borosilicate | Variable | None | Cool to 4 C. Protect | Extract within 14 |
|---------------------------|--------------------|----------|------|----------------------|----------------------|
| Hexachlorobenzene | glass jar with TFE | | | from exposure to | days of sample |
| Hexachlorobutadiene | lined cap | | | light | collection. |
| Hexachloroethane | | | | | Complete analysis |
| Hexachlorocyclopentadiene | | | | | within 30 days after |
| 1,2,4-Trichlorobenzene | | | | | extraction. |

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| | | Minimum | | | |
|---------|-----------|-------------|----------------------|--------------|------------------|
| Analyte | Container | Volume (ml) | Filling Requirements | Preservation | Max Holding Time |

Subcategory I - Polynuclear Aromatic Hydrocarbons

| Acenaphthene | Amber borosilicate | Variable | None | Cool to 4 C. Protect | Extract within 14 |
|------------------------|--------------------|----------|------|----------------------|----------------------|
| Anthracene | glass jar with TFE | | | from exposure to | days of sample |
| Acenaphthylene | lined cap | | | light | collection. |
| Benzo(a)anthracene | | | | | Complete analysis |
| Benzo(a)pyrene | | | | | within 30 days after |
| Benzo(b)fluoranthene | | | | | extraction. |
| Benzo(g,h,i)perylene | | | | | |
| Benzo(k)fluoranthene | | | | | |
| Chrysene | | | | | |
| Dibenzo(a,h)anthracene | | | | | |
| Fluoranthene | | | | | |
| Fluorene | | | | | |
| Indeno(1,2,3-cd)pyrene | | | | | |
| Naphthalene | | | | | |
| Phenanthrene | | | | | |
| Pyrene | | | | | |

Subcategory J - Phthalate Esters

| Butyl benzyl phthalate | Amber borosilicate | Variable | None | Cool to 4 C. | Extract within 14 |
|------------------------|--------------------|----------|------|-------------------|----------------------|
| Bis (2-ethylhexyl) | glass jar with TFE | | | Protect from | days of sample |
| phthalate | lined cap. | | | exposure to light | collection. |
| Diethyl phthalate | | | | | Complete analysis |
| Dimethyl phthalate | | | | | within 30 days after |
| Di-n-butyl phthalate | | | | | extraction. |
| Di-n-octyl phthalate | | | | | |

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XI. Sample Compositing

1.0 Application

1.1 This procedure applies to the compositing of any influent or effluent wastewaters received from an Automatic Sampling apparatus.

2.0 Summary of Method

- 2.1 Generally an autosampler is set up in a field location for up to 24 hours, at which point the sampler unit is returned to the lab for compositing by trained personnel.
- 2.2 When testing for Volatile Organics, it is important to utilize an autosampler equipped with glass vessels and refrigeration capability as it is required during the sampling event.

3.0 Interferences

- 3.1 Inadequate cleaning of the glass vessels of the autosampler and compositing vessel usually account for the majority of the carryover problems.
- 3.2 Periodically run equipment blank from a known clean source to ascertain the effectiveness of the cleaning procedure.

4.0 Equipment and Supplies

- 4.1 ISCO 24 hour autosampler or equivalent
- 4.2 Compositing vessel- glass
- 4.3 graduated cylinders- glass, various sizes
- 4.4 Funnel glass
- 4.5 Acetone
- 4.6 Methanol

5.0 Procedure

- 5.1 Prepare the autosampler for actual setup by critical cleaning of all associated pump tubing and vessels with I lab cleaner followed by solvent rinsing with acetone followed by methanol. It is important that all parts of the autosampler be cleaned adequately due to the ppb levels being tested, and to avoid any contamination of the actual samples. Note that the autosampler should be programmed for adequate purging between samples.
- 5.2 The actual compositing procedure should begin with a visual inspection of all of the vessels to confirm a completely full carousel and adequate volumes with capped vessels.

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- 5.3 When compositing it is important to transfer the same volume of each vessel to a critically cleaned and solvent rinsed glass vessel, capable of holding the composite volume. Typically, 100mL volumes measured with a glass graduated cylinder can be used for a 24 Hour sampling event.
- 5.4 After transferring the individual volumes to the composite vessel, it is important to adequately mix the vessel with no air space due to the nature of the VOC tested.
- 5.5 At this point the representative composite should be carefully dispensed into the respective, properly preserved bottles for metals, VOC's, nutrients etc.
 - 5.5.1 Metals samples are taken in 500 ml HDPE bottles preserved with trace grade Nitric Acid to pH <2.
 - 5.5.2 VOC samples are taken in 40 ml septum capped bottles preserved with ascorbic acid and 1:1 HCl. Add 6 drops of the acid when the bottle is half full.
 - 5.5.3 If testing for TKN, NH₃ or total-PO₄, an HDPE sample bottle preserved with sulfuric acid to pH 2 is utilized.
 - 5.5.4 When testing for NO₂, NO₃, or ortho-PO₄, an HDPE sample bottles with no preservatives should be used. All sample bottles are to be completely full.
- 5.6 All final samples should be refrigerated to 4°C and proper paperwork identifying each bottle should be prepared. Each bottle should be clearly labeled indicating Sample Description, ID number, Date and time sampled, and preservative utilized.
- 5.7 After compositing, the autosampler should be immediately cleaned and prepared for the next sampling event.